## User Manual

# Xcalibur series

Point detector operation

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**Version 1.2**

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oxford diffraction

# Important Information

This user manual applies to the Xcalibur systems manufactured in Poland by Oxford Diffraction. It is a supplementary document to accompany the full Xcalibur User Manual regarding the installation and use of a point detector fitted on an Xcalibur 2 diffractometer.

| | |
|---|---|
| Product: | XCALIBUR |
| Model Type: | PD |
| Electrical Ratings: | 1/N AC 230 V 50/60 Hz 4200 Watts |

## Before attempting to operate the system, PLEASE READ THE INSTRUCTIONS.

This product should only be used by persons legally permitted to do so.

If the equipment is used in a manner not specified in the User Manual, the protection provided by the equipment may be impaired.

## Important Health and Safety Notice

When returning components for service or repair it is essential that the item is shipped together with a signed declaration that the product has not been exposed to any hazardous contamination or that appropriate decontamination procedures have been carried out so that the product is safe to handle.

Care has been taken to ensure the information in this manual is accurate and at an appropriate level. Please inform Oxford Diffraction if you have any suggestions for corrections or improvements to this manual.

Xcalibur service and support is available for technical and operational issues as indicated below.

- **E-mail**: support@oxford-diffraction.com
- **Phone**: +44 (0) 1235 443630 between 8 a.m. and 4.30 p.m. (UK time), Monday to Friday
- **Fax**: +44 (0) 1235 443631

This users' manual has been written according to standard 89/392/EEC and further modifications.

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# 1. Health and Safety Information

## 1.1 General

In normal operation the system is designed to operate safely. All users of Xcalibur should be aware of potential hazards which exist in and around equipment of this type and the ways of avoiding possible injury and equipment damage which may result from inappropriate ways of working. A description of such potential hazards and how to avoid them is given in this section.

This manual adopts the following convention:

**WARNING**
**Indicates a potential hazard which may result in injury or death**

**CAUTION**
**Indicates a potential hazard which may result in damage to equipment**

Warning symbols on the equipment are:

**Protective conductor terminal**

**Earth (ground) terminal**

**CAUTION**
**Risk of electric shock**

**CAUTION**
**Refer to accompanying documents**

**WARNING**
**Radiation Hazard**

See original manufacturers' manuals for further safety data on third party equipment supplied with the system. A list of these is given in this manual.

**WARNING**
**Do not take risks. You have a responsibility to ensure the safe condition and safe operation of equipment.**

**WARNING**
**Xcalibur should only be operated and maintained by authorised operators of the system. An authorised operator is a person who has undergone specialist radiation training and has been trained in the use of Xcalibur by Oxford Diffraction personnel.**

## 1.2 Electrical Safety

In normal use the user is protected from the dangers associated with the voltage, current and power levels used by the equipment. Only personnel qualified to work with the voltages and currents used by this equipment should attempt to disconnect, dismantle or modify the equipment.

### 1.2.1 Potential Electrical Hazards

The following list is not intended as a complete guide to all the electrical hazards on the system, but serves to illustrate the range of potential hazards that exist:

- electric shock
- electric burn
- fire of electrical origin
- electric arcing

### 1.2.2 Recommended Precautions

**WARNINGS**
**All of the electrical equipment supplied as part of the system should be provided with a protective ground. Do not remove protective grounds as this may give rise to an electrical safety hazard. It is vitally important that the system is properly grounded at all times.**

**Follow local and national electrical regulations and procedures.**

**Do not defeat interlocks, remove connectors, disconnect equipment, open safety covers, dismantle or modify equipment unless you are qualified and authorised to do so and you are fully conversant with its operation and potential hazards, or have total assurance through your local electrical permit to work system that the equipment has been made safe.**

**Ensure that the mains supply is fused at an appropriate rating, or fitted with a circuit breaker, and that it can be isolated locally via a clearly labelled, clearly visible and easily accessible isolating switch. Isolate the supply before carrying out any maintenance work.**

**Do not touch any unshielded wires or connectors while mains power is supplied to the system.**

**Do not allow water or any other foreign objects to come into contact with Xcalibur's electrical components.**

### 1.2.3 First Aid

A course in first aid to include methods of artificial respiration is recommended for those whose work involves equipment that may produce a high voltage.

**WARNING**
**Do not attempt to administer first aid to someone who may have suffered electric shock until the source of the shock has been isolated.**
**Mains voltages are present in the system. High voltages are used by the X-ray tube and power supply. These can cause serious injury or death.**
**Only personnel qualified to work with high voltages and currents should perform service or maintenance work on such equipment.**

## 1.3 Mechanical Handling Safety

**WARNING**
**Lifting points are provided for safe handling of components and safe handling practice must be observed to comply with local regulations.**
**Check that lifting points are used only for the job intended.**
**The system itself and some components are heavy and require careful handling. Use safe lifting procedures for heavy items to prevent possible strain injury.**

## 1.4 Safe Mechanical Practice

In normal use personnel are not required to undertake mechanical work. However, servicing or repair may necessitate access to any part of the system. Only personnel who have been trained by Oxford Diffraction to carry out service work on this equipment should attempt to dismantle, modify or repair the equipment.

Water connections should be made and tested in accordance with any local and national safety regulations.

## 1.5 Moving Parts

There are a number of moving parts in the system which are powered by electric motors.

**WARNING**
**Injury could result if clothing or body parts become caught in moving mechanisms.**
**Keep clothing, hands and body parts away from moving mechanisms.**

## 1.6 X-ray Radiation

**WARNING**
**This equipment contains an X-ray tube. Ensure that safe working practices relating to radiation are employed. Follow any local, national or international rules and guidelines.**
**Intentional or reckless misuse of the X-ray generator or its safety devices including safety interlocks and cabinet shielding can result in serious injury or even death.**

During operation, there is an acceptable level of X-ray radiation as based on the recommendations on risk published by the International Commission of Radiological Protection (ICRP) and endorsed by the National Radiological Protection Board (NRPB) in the UK. For use in the UK, the Ionising Radiations' Regulations 1999 should be adhered to. For countries outside the UK the appropriate laws apply such as registration and inspection.

Customers should be aware of their duty of safety to their employees and visitors.

**WARNINGS**
**To prevent injury to personnel and possible damage to the equipment, please note the following guidelines:**

1. **Only authorised personnel who have received appropriate instruction and are aware of the laboratory rules that govern the use of this type of system should operate the system.**

2. **Never dismount the beam stop when the system is operational.**

3. **Do not operate the system without the collimator, unless performing the beam alignment procedure.**

4. **Use appropriate X-ray detection equipment to perform regular radiation checks as per any laboratory rules**

**Use only genuine firmware X-ray tubes, X-ray generators, monochromators, goniometer heads and collimators, as recommended by your Xcalibur supplier. Use of other products may compromise the performance of the shielding and safety system, and may invalidate your warranty.**

# 1.7 Extreme Temperatures

**WARNINGS**

1. **Systems fitted with the low temperature option use liquid nitrogen and/or liquid helium as a coolant. Liquid nitrogen and liquid helium are cryogenic liquids and can cause cold burns. Wear gloves when handling cryogenic liquids and use eye protection. Refer to the information supplied with the equipment for more information.**
2. **During operation the X-ray tube becomes hot. In normal use they are located inside a cabinet and hot parts are not accessible. During maintenance periods, however, it may be necessary to override the interlock so that adjustments can be made. Therefore great care must be taken to avoid touching the X-ray tube when it is operating and for a period of 20 minutes after operation.**

# 1.8 Vacuum

**WARNING**
**When handling and using X-ray tube, particular care should be taken to avoid injury caused by possible implosion of the vacuum tube. Wear eye protection.**

# 1.9 Hazardous or Toxic Materials

Beryllium and beryllium oxide are toxic materials. Follow appropriate handling, shipping, use, storage and disposal procedures and regulations. Refer to BrushWellman Material Safety Data Sheet No. M10 for further information.

**WARNING**
**If Beryllium is exposed to fire, it may oxidise to highly toxic beryllium oxide powder. Do not attempt to clear up the remains of any fire, but contact the relevant local agency stating that there is an incident involving possible beryllium or beryllium oxide contamination.**

# 1.10 Modifications and Service

The manufacturer will not be held responsible for the safety, reliability or performance of the equipment unless assembly operations, extensions, re-adjustments, modifications and repairs are carried out only by persons authorised by the manufacturer. It should be stressed that those parts of the equipment which are interchangeable, and which are subject to deterioration during operation, may significantly affect the safety of the equipment.

# 2. Normal Operation Using a Point Detector

This section of the manual describes the installation and operation of a point detector on an Xcalibur diffractometer fitted with a universal theta arm. For information regarding the use of a CCD detector, refer to the main Xcalibur operators manual.

## 2.1 Installation of the point detector

If a CCD camera is currently installed, determine the orientation matrix of the standard crystal before removing the CCD camera from the diffractometer (this will save a lot of time): Mount the cubic test crystal (either $CaF_2$, or $KAl(SO_4)_2 \cdot 12H_2O$ for example) and optically align it. Carry out a short data collection (e.g. unit cell in 5 minutes) and issue the peak hunting command (**PH S**). Find the orientation matrix and unit cell (**UM F**). Index the cell (**UM I**) and save it by typing **WD T** and giving it a file name. If further information is required about the operation of a CCD camera, refer to the XcaliburCCD manual.

1. Switch off the CCD camera by turning the key anticlockwise on the front panel of the KMW200CCD chiller. In the pull down menu: tools/setup file of the CrysAlis CCD (and CrysAlis RED) programs, swap the setup file from the CCD *.par file to the one relating to the PD (also see next point).

2. Setup file preparation: In the case of a new system, the setup file needs to be checked and altered if necessary. Browse for the setup file using windows explorer. Make a copy of the setup file and save it to disk for backup purposes. Open the original setup file with notepad, scroll down the file and, if necessary, change the last two parameters in the following line of text from:

   GONIOMETER TYPE KUMA_KM4NEW TOP 15.00000 3.00000
   BC2_SAPPHIRE FALSE 1.00000 1.00000 1.00000 TIME FALSE FALSE
   TRUE **TRUE FALSE**

   To:

   GONIOMETER TYPE KUMA_KM4NEW TOP 15.00000 3.00000
   BC2_SAPPHIRE FALSE 1.00000 1.00000 1.00000 TIME FALSE FALSE
   TRUE **FALSE TRUE**

3. Restart the CrysAlis CCD (and CrysAlis RED) programmes

4. Drive the goniometer to theta = 90 deg (**GT T 90**), and drive the camera distance to 60 mm (**GT D 60**).

5. Unplug all the connectors to the CCD head, note which fibre optics cable is connected to which socket; mark them if necessary to save time when reconnecting. The order of the water pipes is not important.

**CAUTION**
**Take care when disconnecting water pipes to prevent drips of water landing on the camera.**

6. Unscrew the two upper M4 screws connecting the slider to the lead screw which defines the camera distance. Turn the end plate of the slider anticlockwise by 180 degrees. Remove the CCD camera from the slider carefully and place in the storage box. The empty slider will appear as in Figure 2.1

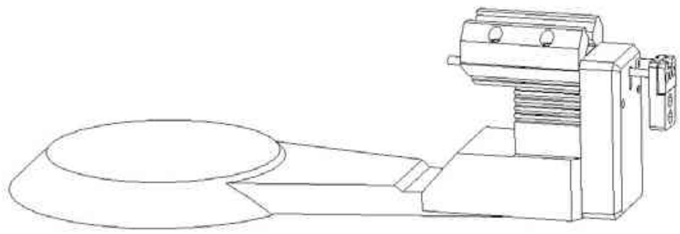


***Figure 2.1 The universal theta arm***

**CAUTION**
**Take care not to allow the CCD head to collide with the beam stop or kappa block as the slider has very low friction.**

**WARNING**
**IMPORTANT! Switch the interface off at this point using the power switch on the front of the interface before the high voltage cable is plugged into the point detector. This cable could carry a voltage of up to 1000V.**

7. Replace the CCD camera by the point detector.

8. Turn the end fixing plate clockwise by 180 degrees and fix the PD slider into place using the two screws that were removed in step 6. Connect the PD interface cable and screw in the fixing screws to prevent accidental disconnection.

**WARNING**
**The point detector interface cable carries a high voltage**

9. Switch the interface back on and issue the command **GON REINIT**. Check that no error messages appear. Drive all angles back to zero (**GT a 0 0 0 0**).

10. In the case of a new machine, set the detector distance to 130mm (**GT D 130**) and open the cabinet door. Drive theta to 90 degrees (**GT T 90**) and -90 (**GT T -90**) and check that there are no potential collisions with the cabinet. Move the goniometer if necessary.

11. Issue the command **TY P** to display the data collection parameters in a table in the history window. Check and change if necessary the following parameters:

| | |
|---|---|
| **SC S 0.15** | Omega scan speed |
| **SC W 1.3 0.35 1.008** | Scan width |
| **SC T 0 2** | Type of scan |
| **MO B 0.5** | Background mode |
| **MO S 1 61 3 25 0.015** | Scan mode |
| **DA 1.33 1.33** | Detector aperture for the typically used slits |
| **DL 2 100** | Discrimination level |
| **SW CE 1.5** | Centering conditions |
| **SW SMI 0** | Mode of operation for SM **I** (0.01 for synchrotrons only) |
| **TR 2 60** | Theta range |
| **FI 100000** | Filter setting |
| **HV 170** | Counting chain high voltage level |
| **GA 200** | Counting chain gain level |
| **LL 30** | Counting chain low level setting |
| **WI 170** | Counting chain window setting |

12. In the pull down menu tools/options check and set to zero all the correction factors except for: alpha and beta (which should be the same as the CCD setting), detector distance (which should be 130) and the X and Y positions of the detector (which should both be the theoretical values of 512). Don’t recalculate the peak table (if any) on exit.

13. If the orientation matrix and unit cell are unknown find them using peak hunting (for example: **PH S 25 10 20 -60 -20 0 359**).

> **NOTE**
>
> **to find a cell more rapidly, it may be better to increase the size of the slits from the front of the detector. If you do this, you must type the command DA X Y to update the new detector aperture setting (where X and Y are the slit size)**.

14. Once the data collection has finished issue the command **UM F** (or **UM C**) to find the unit cell then **UM I** to index it.

> **NOTE**
>
> **at this stage the unit cell will be of poor quality due to the lack of calibration.**

15. If the orientation matrix is known, read the peak table from disk if needed using the command **RD T**. If the data were from a CCD data collection, use the command PT **ANGLES** to calculate the setting angles.
16. Update the peak table using the command **UM U**. This will cause the diffractometer to search for all of the theoretical peaks in the peak table and will take about 20 minutes to refine 20 peaks.
17. Save the final model by issuing the command **WD CAL.** A backup file will be written at the same time. Repeat the **UM U** procedure to update the peak table. If the standard deviations for the unit cell lengths are less than 0.001, the model is finished.

If the model is insufficient, further corrections must be carried out. This may be in the case of a new machine or a machine that has undergone extensive adjustments. The following additional procedure should then be followed:

1. Issue the command **PT E** to examine the peak table. Click on the radio button in the coordinates section at the bottom of the window labelled angles. Find a strong reflection.
2. After finding the strong reflection, replace the 1.33 slits (if they were previously removed) and type **DA 1.33 1.33**. Centre the reflection by typing **CE**. Check the counter line electronics characteristics by issuing the command **SM C 170 200 10 200 1 1**. Repeat this if necessary changing HV and/or GA and if necessary adjust LL (value just to the left of the peak) and WI (value just to the right of the peak) parameters. To examine the profile of the peak type **SM S 30 0.5**. Save the new parameters by typing WD CAL.

To find the initial zero correction for theta and horizont:

3. If the goniometer is not already set at the reflection position, type **GT R h k l** (where h k and l are the coordinates of the reflection that was chosen in point 2). Find the initial zero

correction for theta and horizontal using the **ZC A** command. This command will examine the peak from two opposite directions and will output a suggested correction in the history window. Apply the suggested correction by copying the line of text from the history window and pasting it into the command line and pressing return. Re-issue the **ZC A** command and repeat this procedure approximately 5 times until there is little difference between the current and suggested correction (you may type **TY P** to see the current setting). Note that if the corrections are very large or the reflections are very narrow, **ZC A** may fail on the first attempt. In this case, use larger slits and/or wide centring parameters (e.g. **SW CE 2**).

> **NOTE**
>
> **before reissuing the ZC A command, you must GT R h k l (or GT A o t k p) to recover the starting reflection position.**

To calculate the zero corrections for omega and kappa:

4. Build a peak table containing 24 reflections of the 3 3 5 family (for Mo radiation) or the 2 2 4 family (for Cu radiation): Type the command **GT R 3 3 5** then **PT A** (this adds the current goniometer setting to the peak table as a peak), **UM I** (to index the new peak), then **PT E** (to open the peak table editor). Note that **UM I** will fail if the peak table contains fewer than 3-4 reflections. Erase all but the last added reflection and click on exit. Issue the command **PT L 12** (this will add peaks to the peak table which are related by the Laue symmetry to the 3 3 5 reflection. Issue the command **PT E** to examine the peak table. Click on the radio button at the bottom of the window in the coordinates section labelled angles. Sort the reflections by descending kappa (by clicking at the top of the kappa column). Remove any reflections with a kappa angle of less than 2 degrees as the centring process will be very slow for these reflections. Click on the exit sorted button. Save the peak table using the command **WD T**.
5. Calculate the initial zero corrections for omega and kappa by typing **ZC T**. The suggested correction will be typed into the history window. Issue the command **TY P** to display the current values. Copy and paste the **ZC S** line from the table in the history window to the command line and modify the 1.st and 3.rd value by adding approximately 90% of the suggested change.

> **NOTE**
>
> **Check that the suggested change is a sensible value**

6. Repeat points 4 and 5 approximately 5 times until the suggested changes are negligible.
7. Issue the command **REFINE MODEL.** Set the cell type to LAT_AAA and ANG_909090. In each cycle check only ONE radio button at a time to refine in order: alpha, beta, kappa and omega. Refine theta separately otherwise there is a risk that, because the command

was originally designed to deal with CCD data, the refinement may become unstable due to the small number of observables.

8. Note the calculated corrections for omega, theta and kappa and add the new values to the parameters by issuing the command **ZC S do dt dk**

**NOTE**

**the only way to measure the correction for horizont (4.th parameter) is ZC A.**

9. Using tools/options reset the corrections for omega theta kappa and phi to zero - don't recalculate the peak table on exit from the panel.
10. Save the final model by issuing the command **WD CAL**. A backup file will be written at the same time. Repeat the **UM U** procedure to update the peak table. If the standard deviations for the unit cell lengths are less than 0.001, the model is finished.

## 2.2 Removal of the point detector

1. Drive the goniometer to theta = 90 deg (**GT T 90**), and drive the camera distance to 60 mm (**GT D 60**).

**WARNING**

**IMPORTANT! Switch the interface off at this point using the power switch on the front of the interface before the high voltage cable is plugged into the point detector. This cable could carry a voltage of up to 1000 V.**

2. Unscrew the PD interface cable fixing screws and disconnect the PD interface cable.

3. Unscrew the two upper M4 screws connecting the slider to the lead screw which defines the camera distance. Turn the end plate of the slider anticlockwise by 180 degrees.

**CAUTION**
**Take care not to allow the point detector head to collide with the beam stop or kappa block as the slider has very low friction**

4. Place point detector in storage case. The point detector interface cable may be left in place but should be fastened securely to prevent the connector colliding with another detector, the goniometer, beam stop or crystal.

# 3 General Commands

## 3.1 Gt - Goto Angles Commands

When Xcalibur is not collecting data the goniometer axes can be driven to accessible positions using the following commands:

| | |
|---|---|
| **gt a** om th ka ph | go to angles with values omega (om), theta (th), kappa (ka) and phi (ph) |
| **gt o** om | go to omega angle 'om' |
| **gt t** th | go to theta angle 'th' |
| **gt k** ka | go to kappa angle 'ka' |
| **gt p** ph | go to phi angle 'ph' |
| **gt d** det | go to detector distance 'det' in mm |
| **gt r** h k l | go to a reflection with specific h k l value |

## 3.2 Ty – Type Details Commands

The ty command allows the user to print a variety of current settings to the history window:

| | |
|---|---|
| **ty p** | print current Xcalibur parameter settings |
| **ty z** | print the current zero correction parameters |
| **ty u** | print current UB matrix |
| **ty l** | print current unit cell and lattice settings |
| **ty t** | print current contents of peak table |

## 3.3 Single measurements

| | |
|---|---|
| **sm i** time repeats | stationary intensity measurement with exposure time (time) and number of repetitions of the measurement (repeats) |
| **sm r** h k l psiang psistart psistep | scan a reflection with the given h k l values with the optional parameters: psiang (psi angle for |

| | |
|---|---|
| | the scan), psistart (starting psi angle for a sequence of scans), psistep (psi step angle for measuring a sequence of scans) |
| **sm s** steps time | step scan with a number steps (steps) and an exposure time (time) |
| **sm ao** om th ka ph scanwidth time | Single measurement omega scan with angles<br><br>om, th, ka and ph are the omega, theta kappa and phi settings in degrees<br>scanwidth = degrees<br>time = seconds |
| **sm eta** starteta step steps time | step scan moving every reflection vertically in the aperture window<br><br>starteta = starting eta value. Actual goniometer position is assumed to be theta 0.0.<br>step = scanstep in deg.<br>steps = number of steps for scan<br>time = exposure time in sec per step |
| **sm help** | help overview of sm commands |
| **sm i time** | single static measurement of exposure time in seconds |
| **sm rp** | 30 secs phi rotation photo |
| **sm o** startangle scanwidth time | omega scan |
| **sm p** startangle scanwidth time | phi scan |
| **sm q** h k l st #s1 sw1 u1 v1 w1 [#s2 sw2 u2 v2 w2] [filename] | records a q scan<br><br>H K L = indices of selected peak<br>st = measurement time of one scanning point [sec]<br>#s1 = number of scanning points in the [U1 V1 W1] direction<br>sw1 = increment of scanning angle for [U1 V1 W1] direction [Deg]<br>U1 V1 W1 = indices of the first scanning direction<br>#s2 = number of scanning points in the [U2 V2 W2] direction<br>sw2 = increment of scanning angle for [U2 V2 W2] direction [Deg]<br>U2 V2 W2 = indices of the second scanning direction<br>[filename] = optional filename. |
| **sm r** h k l [psistart [psiend psistep]] | measure a single reflection<br><br>h k l = reflection index, may be fractional.<br>psistart = optional psi angle.<br>psiend = optional psi start angle for measuring a sequence.<br>psistep = optional psi step angle for measuring a |

| | |
|---|---|
| | sequence |
| **sm t** startangle scanwidth time | theta scan |

# 3.4 Commonly used unit cell/Indexing commands

| | |
|---|---|
| **um f** [lengthdeviation angledeviation fractionindexed] | Automatic unit cell determination (indexation, refinement, reduction)<br><br>[lengthdeviation angledeviation fractionindexed] = The defaults are 0.05, 0.1, 0.7. You can loosen this condition in case of an unsuccessful indexing: typically 0.3 0.3 0.5. |
| **um i** [indexrejectioncriterion] | Index and refine unit cell<br><br>[indexrejectioncriterion] = rejection criterion which determines the maximum allowed deviation for which a reflection is considered indexed. |
| **um c** [change #]\|[c11 c12 c13 .. c31 c32 c33] | Change orientation matrix<br><br>[change #] = transformation number from table obtained by typing um c: [c11 c12 c13 .. c31 c32 c33] – direct space transformation matrix |
| **um reduce** | Apply Niggli reduction of unit cell |
| **um r** [symmetrycode] | refine UB under symmetry constraint<br><br>[symmetrycode] – The necessary code can be obtained by just typing um r |
| **um sarray** arraynum ub11 ub12 ub13 .. ub31 ub32 ub33 | Store the defined ub unit cell matrix to a buffer array. 8 available. Used to define a number of ub matrices for data reduction of twinned data.<br><br>Arraynum (0..7) storage buffers for unit-cell matrices<br>Ub11 … ub33 unit-cell ub matrix |
| **um u** | activates the procedure to refine the crystal orientation automatically |

# 3.5 Peak table commands

| | |
|---|---|
| **pt clear** | clearing of the peak table |
| **pt a** | add a peak to the peak table |
| **pt e** | Peak table edit |
| **pt expand** n mmin mmax intmin dmax dmin | Peak table expand<br><br>n = number of reflections required<br>mmin = min order of difference -1 for CCD (-3 Point detector)<br>mmax = max order of difference +1 for CCD (+3 Point detector)<br>intmin = Intensity threshold<br>dmax = max d spacing<br>dmin = min d spacing |
| **pt l** lauecode | add reflections to the peak table according to their Laue symmetry<br><br>lauecode = The following codes can be selected:<br>1 : -1<br>2 : 2/m<br>3 : mmm<br>4 : 4/m<br>5 : 4/mmm<br>6 : -3<br>7 : -3m1<br>8 : -31m<br>9 : 6/m<br>10 : 6/mmm<br>11 : m-3<br>12 : m-3m |
| **pt sa** settingno | add reflections from a different setting position<br><br>settingno = The settingno refers to the basic measurement settings. |

# 3.6 System commands

The following commands may be issued to access the Windows system operations:

| | |
|---|---|
| **System dos** | spawns a MSDOS window with the current directory path being used in the CrysAlis program. |
| **System explorer** | spawns an Explorer window with the current directory path being used in the CrysAlis program |

# 3.7 Writing to disk

Current machine parameters, images and the contents of peak hunting tables can be written to disk

| | |
|---|---|
| **wd p** | write disk parameter settings. Saves the current machine parameters to disk |
| **wd ph** | write disk peak hunting. Saves the current contents of the peak hunting table to disk |
| **wd t** | write disk table. Saves the current contents of the peak table to disk |

# 3.8 Reading from disk

Machine parameters, images and peak tables can be read from disk using the following commands:

| | |
|---|---|
| **rd p** | read disk parameter settings. Reads stored machine parameters from disk |
| **rd ph** | read disk peak hunting. Reads a stored peak hunting table from disk |
| **rd t** | read disk table. Reads a stored peak table from disk |

# 3.9 Exiting the CrysAlis CCD program

To exit the CrysAlis CCD program the command **en** should be issued. This drives the goniometer axes to their home zero positions and exits the CrysAlis CCD program.

# 4 Standard Point Detector Experiment

In order to use the point detector (PD), the correct parameter file must be loaded. This is due to subtle changes between the layout of the GUI for the CCD experiments and the PD experiments. The location and type of parameter file may be checked by using the tools/setup file pull down menu. If the setup file is changed, the programme must be restarted in order to load this new parameter file.

> **NOTE**
>
> **It is important to make sure the setup file is correctly formatted for point detector operation and not for CCD operation otherwise data collection will not be possible. See section 3.5.1 for details.**

A standard crystallography experiment using a point detector consists of 7 main steps:

1. Crystal mounting and alignment
5. Peak hunting
6. Unit cell determination
7. Data collection
8. Data processing
9. Space group determination
10. Structure solution and refinement

The procedure for a standard experiment follows:

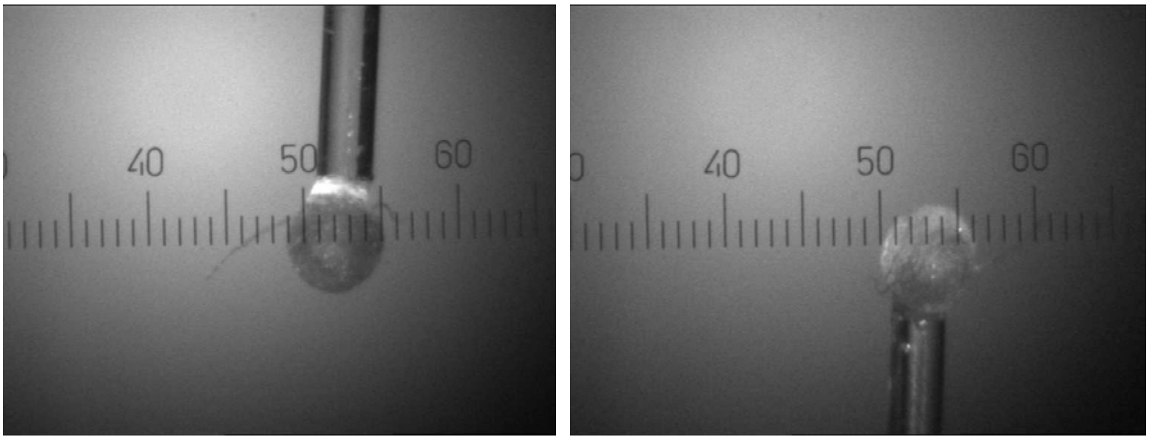
## 4.1 Crystal Mounting and Alignment

> **Caution**
>
> **Press 'STOP' on the remote control or 'Ctrl' on the keyboard to stop movement of the equipment in an emergency. Mechanical movement of the goniometer and CCD detector may be performed using the remote control.**

1. Start the CrysAlis CCD application
2. Press **F12** key to release control from the computer to the remote control unit
3. Press the 0 and HOME buttons on the remote control to drive the goniometer angles to the zero / home position.

4. Mount the xyz goniometer head with crystal attached to, for example a glass fibre or nylon loop with oil or glue.
5. Press Lower and 0 on the remote control. This will drive the goniometer to the correct orientation to allow optical alignment of the crystal.

> **NOTE**
>
> **The settings lower and upper refer to the glass stick position on the video monitor**




Upper Setting Lower Setting

*Figure 4.1 Optical alignment of the crystal*

6. Use the tool provided with the goniometer head to adjust the vertical height and horizontal position of the crystal, such that the crystal is in the centre of the video monitor screen.
7. Press **180** on the remote control to rotate the crystal through 180 degrees. If the crystal's horizontal position has moved on rotation adjust the position. Press **0** and repeat this procedure until rotation gives no movement of the crystal.
8. Repeat the above process, rotating between **90** and **270** degrees.
9. Press **Upper** on the remote control. The goniometer will now move to the upper position such that the goniometer head is located behind the collimator. If the vertical height of the crystal has changed, adjust and return to the lower position. Repeat until the vertical position is unchanged between the upper and lower positions.
10. Press **Lower** and check alignment of the crystal on rotation between 0 and 180, 90 and 270 degrees.
11. Press **0** and **Home** to return the goniometer to its zero position.
12. Exit alignment procedure by selecting **OK** on the computer screen. This will return goniometer control to the computer and prevent use of the remote control.

## 4.2 Setting the data collection parameters

- **Two theta range**

The two-theta range may be set by the command **TR tthmin tthmax** (where TR = theta range, tthmin is the minimum value of 2 theta and tthmax is the maximum 2 theta value) typical values are: **TR 4 60** or **TR 4 80** for Mo radiation.

- **Scan width**

The scan width depends on the type and quality of the crystal. The sc w command sets the scan width parameters: **SC W Oa Ob C** where Oa = scan width in omega ($0.0 \le Oa \le 180$), Ob = scan widening parameter ($0.0 \le Ob \le 2.0$) and C = scan centre shift parameter ($1.0 \le C \le 1.1$)The scan width increase parameter and scan centre shift are often left unchanged, typically: **SC W 1.2 0.35 1.0008**

> **NOTE**
>
> **The Oa and Ob parameters of the SC W command determine the omega scanwidth. The total scanwidth is calculated according to the formula: Oa + Ob ´ tan (theta).**
>
> **The last parameter allows to adjust the scan centre to any position between Ka1 and Ka2 peaks. The position of a reflection obtained from the orientation matrix corresponds to the Ka1 peak position i.e. Bragg angle Ta1. The scan centre can be changed by calculating new theta angle according to the formula: sin(theta) = C * sin(Ta1).**

- **Scan type**

The scan type command **SC T (Tx) (f)** determines the type of scan to be made. Tx = the threshold angle ($0.0 \le Tx \le 90.0$) and f = the multiplier for theta motor ($0.0 \le f \le 2.0$). Omega scans are made for reflections with the theta Bragg angle less than Tx. Omega-theta scans are made for reflections with the greater Bragg angle. The scan type is usually set to Theta-Twotheta: **SC T 0 2**

> **NOTE**
>
> **The theta motor speed is calculated as f * Vo and it must be inside the same range as the omega scan speed, (sc s).**

- **Scan speed**

The scan speed is dependent upon the crystal quality and the requested quality of data. The sc s command **SC S (Vo)** sets the omega scan speed in deg/sec. The ranges for Vo are $0.004768 \le Vo \le 1.5$ For routine organometalic data collections use: **SC S 0.15**

- **Mode of scan**

The mode of scan command **MO S m s n1 [n2 Vmin]** specifies the type of scan to be made during data collection. The parameters are: **m** - mode of scan ($0 \le m \le 2$; 0=BPB scan, 1=continuous-integrative step scan and 2=stationary step scan); **s** - number of scan steps (not used for m=0) ($10 \le s \le 1024$); **n1 and n2** minimum accepted **I**/sigma(**I**) and requested **I**/sigma(**I**) ($-3 \le n1 \le n2 \le 50$) and **Vmin** - Minimum rescan speed in deg/sec

($0.004768 \le Vmin \le 1.5$). The most typical mode of scan is an integrating stepscan with prescan. This skips reflections that have I/Sigma(I) values worse than 3.0, whilst trying to obtain I/Sigma(I) ≤25. This should give an R1 factor of the refined structure ≤5% For example:

**MO S 1 55 3 25 0.015**

or

**MO S 1 60 1 10 0.0050**

**NOTE**

**m=0**

**A continuous scan according to the parameters given by SC S , SC W and SC T commands will be made. In this case the parameters must be omitted. For n1 = n2 parameters n2 and Vmin may be omitted also.**

**m=1**

**An integrative step scan according to parameters given by SC S , SC W and SC T will be made . The total *scan width* is divided into s steps and for each step a continuous scan is made.**

**m=2**

**Is equal to mode = 1 with an exception that a stationary measurement of each centre of step is made. The step measuring time is determined by the step width and the scan speed Vo, and is equal to step width/Vo.**

**In all scan modes the attenuation filter is automatically inserted when the counting rate is higher than the level set by FI and scans are repeated.**

- **Background mode**

The mode of background calculation is set by the command **MO B f** (0.01 <= f <= 2.0). It is usually set to 0.5, **MO B 0.5,** which means that 50% of scan steps (25% from each side) is used to calculate background. This must be taken into account when choosing the scan width.

**NOTE**

**For MO S 0 the background is measured stationary at each side of the scan during time determined by**

**t = f*scantime/2 .**

**The total background time is twice the given value. For MO S 1 and MO S 2 the parameter f determines the division of the measured profile into reflection peak and background regions. The left and right backgrounds include the same number of outer profile points : s1 = 0.5*s*f/(f+1) ,**

| |
|---|
| **where s is the total number of scan points.** |

- **Index limit**

The index limit command **IL hmin hmax kmin kmax lmin lmax** sets the index limits for the data collection. The range is dependent upon the unit cell lengths. Signs are dependent on the crystal system (unique reflections set) and on the crystal orientation. If possible, the kappa axis should be on the negative side during data collection. Typical values are: **IL 0 10 0 10 - 0 0**

- **Reference reflections**

Reference reflections are particularly important in point detector data collections where the long data collection time may allow for crystal decay or movement. The RR command:

**RR rr_num [interval om_tol int_tol [h k l at [h k l at [h k l at]]]]**

specifies the reference reflections and conditions for initialization of the reorientation procedure.

- **rr_num** (number of reference reflections: 0≤rr_num≤3) and if rr_num>0: **interval** (interval between reference reflections measured: 1≤interval≤32000)
- **om_tol (**requested omega repeatability: 0.0025≤om_tol≤10)
- **int_tol** (allowed intensity fluctuations parameter (number of sigmas by which a fluctuation triggers the re-centring): 1≤int_tol≤100)
- **h k l** (Miller indices of ref. reflection:-126≤h,k,l≤126)
- **at** (attenuation filter use: 0,1 (0 = filter out, 1 = filter in)).

Up to 3 reference reflections may be measured during data collection. When the number of reference reflections n1 is equal to zero the further parameters should not be entered. The interval between two successive measurements of reference reflections is specified by the given number n2 of measured reflections. The reference reflections are measured with a step scan according to parameters given by **SC W**, **SC T** commands and a step number specified by n3. The step measuring time and the attenuation filter setting are determined for individual reflection by t and fil respectively. For each reference reflection one has to specify its Miller indices h,k and l which you choose from the peak table which was recorded during the peak hunting procedure. They should be strong, but not need an attenuator. Typically they are measured every 50 - 100 reflections, for example

**RR 3 100 0.15 12 1 2 3 0 2 3 4 0 3 4 5 0**

- **Reflection conditions**

The reflection conditions command **RC** allows the user to select which reflection conditions are applied during the data collection due to the centred cells, glide planes and screw axes (For further explanations see "International Tables for Crystallography" (1983), edited By Theo Hahn, Vol A, pp. 27-29 and 41-47, Dordrecht (Holland) / Boston (U.S.A): D. Reidel Publishing Company). Typically all reflections should be measured, just in case the *a-priori* assumption of lattice centring or space group etc. is wrong. It is possible to skip extinct reflections by using the RC command. It will display a menu allowing the extinction rules to be set. These extinction rules are not saved with the parameters so have to be input at the start of each data collection. This is because these rules may easily be overlooked (after typing parameters from the previous data collection on screen) causing the measurement of an incomplete reflections set. There is also a

'negated' option, allowing the measurement of, for example, forbidden or mistakenly skipped reflections.

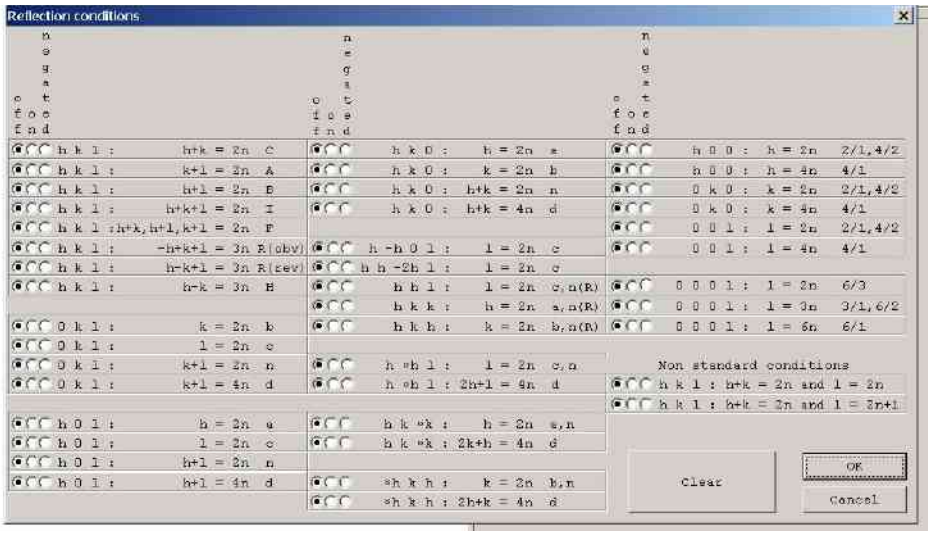



*Figure 4.2 The reflections conditions programme*

> **NOTE**
>
> **One has to be very careful not to create contradicting extinction conditions (the conditions are always logically interpreted by "and").**

- **Order of index change**

The order of index increase may be set using MA S command: **MA S n1 n2 n3** (where n1, n2, n3 – define the order in which the indexes change (first-second-third) and 1=h,2=k,3=l)
Typically one would use

**MA S 3 2 1**

- **Setting type**

Typically, the setting type should be chosen before peak hunting, but it is also possible to do it here, for example

**SW S 1**

- **Slits**

The appropriate slits must be set

**DA 2.0 0.5** or **DA B 2.0 1.0**

> **NOTE**
>
> **To check the result of choice one can use dc t emulator which prints kappa angle for each reflection.**

# 4.3 Peak Hunting

The detector aperture is determined by two sets of manually exchangeable slits which are inserted in the front of the detector. The inserted combination of slits is expressed in degrees (DH and DV for the horizontal and vertical apertures, respectively).

> **NOTE**
>
> **The detector aperture parameters are vital in the centring, zero corrections and peak hunting procedures. When the DA settings are wrong, these procedures may fail.**

The slits are available in a range of sizes

1. Check the aperture size of both the vertical and horizontal slits. If necessary, adjust the detector aperture parameter setting by typing **DA DH DV** where DH is the aperture of the horizontal slit and DV is the aperture of the vertical slit.

> **NOTE**
>
> **You may use a bigger aperture (e.g. DA 4 2) to speed up peak hunting. Then use smaller slits (e.g. DA 1.33 1.33 depending upon the size of your reflections) and update the peak table before going to the auto-indexing routines to increase the accuracy. This is usually much faster than doing peak hunting immediately using the small slits.**

In the peak hunting procedure an area of the reciprocal space is investigated by phi scans with a scan speed ‘v’ The peak centring procedure will occur when a signal is observed above the discrimination level (N). Set the phi scanning speed and discrimination level parameters using the DL command: **DL v N** (typical starting values for v and N are 3 and 1000 respectively). The value of v can range from 0.01 to 3 (mostly a maximum of 2 is used) and the value for N ranges from 0 to 10000.

> **NOTE**
>
> **If you do not know the parameters, the discrimination level parameter should be set to a high value (e.g. 1000) and shortly after peak hunting has started (< 1 min. typically) the procedure should be interrupted and a threshold 20 - 30% higher than the noise level threshold should be set. The speed parameter should be decreased only in case of weak diffractors.**

2. Select the setting type for operation using the command **SW S setting** (where setting is the setting option). The options for setting are 0 = bisecting mode, 1 = traditional mode and 2 = high pressure mode (‘eularian’, phi at zero). Normally one would choose the bisecting setting: **SW S 0**

During peak hunting, the area is sampled starting at T1, K1 and P1. The omega angle is determined by the standard (traditional) setting requirement. The phi scans oscillate between P1 and P2. At the end of each phi scan the crystal is rotated, increasing the kappa angle. When kappa reaches its maximum value K2, the 2theta angle is changed and kappa is reduced to its minimum value K1 and so on.

**NOTE**

**All peaks found will be appended to peak table, so in the case of a new data collection, any existing peaks should be erased first by using the PT CLEAR command.**

3. Start the peak hunting procedure either by using default values, using the command **PH S,** or by using custom settings by typing the command **PH S n T1 T2 K1 K2 P1 P2**

   - n is the number of reflections to find
   - T1 and T2 are the minimum and maximum values for theta (0≤T1≤T2≤90)
   - K1 and K2 are the minimum and maximum values of Kappa (-130≤K1≤K2≤130)
   - P1 and P2 are the minimum and maximum values for phi (0≤P1≤P2≤360)).

**CAUTION**
**It is recommended to routinely use values of Kappa that are between -70 and 70 to reduce the risk of finding reflections to close to the collision limits of the goniometer.**

4. When a peak is found the centring procedure is carried out and the result is typed into the history window in the following format:

   (No) (omega) (2theta) (kappa) (phi) (Int) (filter setting)

   Where No is the number of the reflection in the peak table, omega, 2theta, kappa and phi are the goniometer settings and Int is the measured peak intensity.

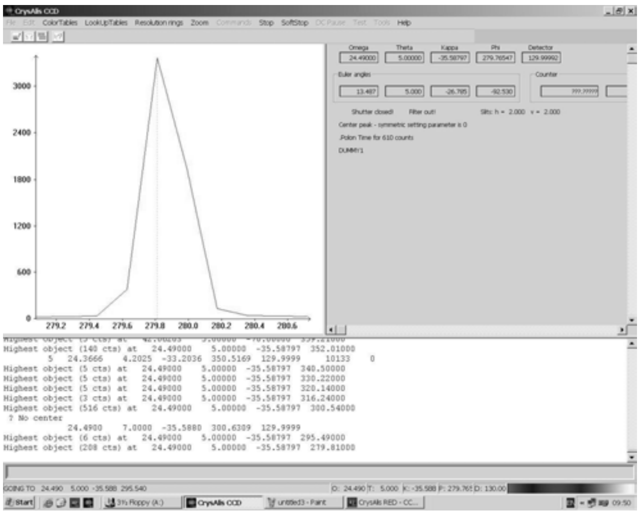


*Figure 4.3 The peak hunting process*

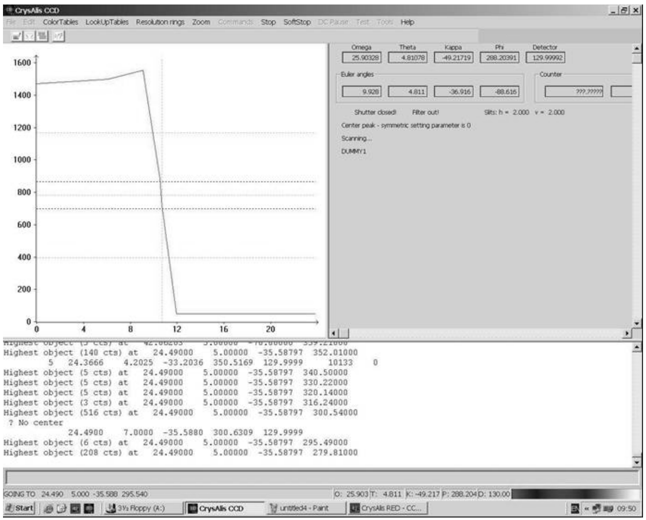



***Figure 4.4 The centring procedure***

5. The top intensity Int is the number of counts recorded in one second. The angles are converted to reciprocal coordinates x(1),x(2) and x(3) and these are compared with the coordinates already stored in the peak table. A new peak is said to be found, when the table does not contain a peak with coordinates y(1) , y(2) and y(3).  When the new peak has been found the coordinates and intensity are added to the end of the peak table. If a duplicate peak is observed, the output contains (=No) instead of (No).When the intensity is greater than that already stored in the table the new peak position is stored instead of the old one. When the search is complete or when the peak hunting procedure is interrupted, the unit cell may be determined.

# 4.4 Unit cell determination

After successful peak hunting:

1. Find the orientation matrix - currently two procedures are available, **UM F** working in reciprocal space and **UM T** in direct space.

2. Once the orientation matrix and unit cell have been found, there are a number of functions which may be utilised:

   - The found orientation matrix may then be refined by issuing the command **UM I**
   - The orientation matrix may be changed in direct space using the command **UM C.** Using this command will cause a number of options to be typed to the history window. Typing **UM C** followed by the number of the desired option will change the orientation matrix. Alternatively, a transformation matrix may be typed after **UM C** to carry out the desired transformation.
   - The orientation matrix may be changed in reciprocal space using the command **UM CREC** in the same way as using the command **UM C**
   - A constrained cell may be displayed by typing **UM R** followed by a Laue symmetry option.
   - **UM REDUCE** will facilitate additional unit cell reduction if required.

3. Once the desired cell and setting have been determined, the peak table and orientation data may then be written to disk using the command **WD T.**

# 4.5 Data Collection

1. If the orientation matrix of the crystal is known, you may import an existing peak table. Alternatively, you may input the matrix using the command line. Then using sequence: **GT R** (go to reflection), **CE 1** (centre reflection) and **PT A** (add to peak table) fill the peak table with approximately 10 reflections, refine the existing matrix and skip to point 4. If necessary - extend the existing peak table.

2. Before starting data collection it is advisable to update the peak table using the command **UM U** (This will cause the diffractometer to search for the reflections in the peak table and re-centre them).

3. To start the data collection procedure, type **DC S**. If you start the data collection without any parameters the program will use minimal values of HKL taken from IL parameter. After interrupting, a data collection may be restarted by typing the command **DC R**. A set of reference reflections will be collected followed by data collection from the next HKL from sequence:

4. When data collection finishes, go to the data processing section.

# 4.6 Data Processing and Reduction

Reflection data collected during the data collection commands need to be converted into standard Shelx *.hklf4 format using the DC REDPD command. Additionally some corrections may be applied - typically spherical absorption or empirical absorption using the shape of the crystal.

1. After invoking the **DC REDPD** command, an option screen appears. It is necessary to select the relevant data collection *.DC1 file by clicking the "File name" button - an open file window will appear.

2. A number of options will be displayed. Typically the information in the data collection file is converted to Shelx format using default parameters. Processing may now be started by clicking on the **process now** button and the output will be in the form of *.hkl, *.sum and *.lst files.

3. If necessary, by clicking "Enable recalculation", all the reflections may be reintegrated and corrected using the following options:

   - **Attenuation factor** – a correction is applied to reflections measured with attenuator on (see AF I command). The allowed range is 1 to 1000.
   - **Back/peak ratio** - allows redefinition of the ratio between the number of scan steps used for background evaluation and the steps integrated as intensity. This means overriding the parameter set by the mo b command. This is the most common reason for needing to recalculate.
   - **Polarisation/Monochromator** – this is useful only in cases when data were collected using the wrong option. The options are: e1-e3-plane (Xcalibur1), e1-e2-plane (MAR-Huber) and synchrotron. Typically e1-e3-plane (Xcalibur1).
   - **d-value of monochromator/Polarization factor** - this is also only useful in cases where data were collected using the wrong option. The allowed ranges are 0.1 to 10000 but typically the value should be 0.98.
   - **Dead time corr/Dead time corr(mon)** - this is also only useful in cases where data were collected using the wrong option. The ranges are 0 to 1 but typically the value should be $1.7 \times 10^{-6}$ for both of these parameters.

- **Cut off multiplier** – This will clean the output file by removing reflections below a certain I/sigma value. Tick the radio button to apply the cut-off and click on the box to edit the limit.

Data may also be corrected for absorption**:**

- **Abs. for empirical shape - EDIT/IMPORT SHAPE** – this allows the user to apply a correction according to the shape defined using the **abs display** function. Click on the radio button to apply the correction and click on the box to open the absorption correction module and import the crystal shape model.
- **Abs. for sphere correction (μr)** – this allows the user to apply a spherical absorption correction. Click on the radio button to apply the correction and click on the box to edit the absorption coefficient of the crystal.

**NOTE**

**The "Screen output" option can only be used in debug mode, and should be switched off. The reference reflection correction is on by default and rescales data collection for decay of crystal comparing intensities of reference reflections.**

4. The hkl file format may be chosen. The options are: SHELX hkl F2 s(F2) b, SHELX hkl F2 s(F2) b dircos (which includes the direction cosines) and SHELX hkl F2 s(F2) psi schwaba (which includes Schwarzenbach psi information for use with the **SCALE3 ABS** command). By default the SHELX hkl F2 s(F2) b output format is selected.

5. Clicking "Process now" will start the data processing operation.

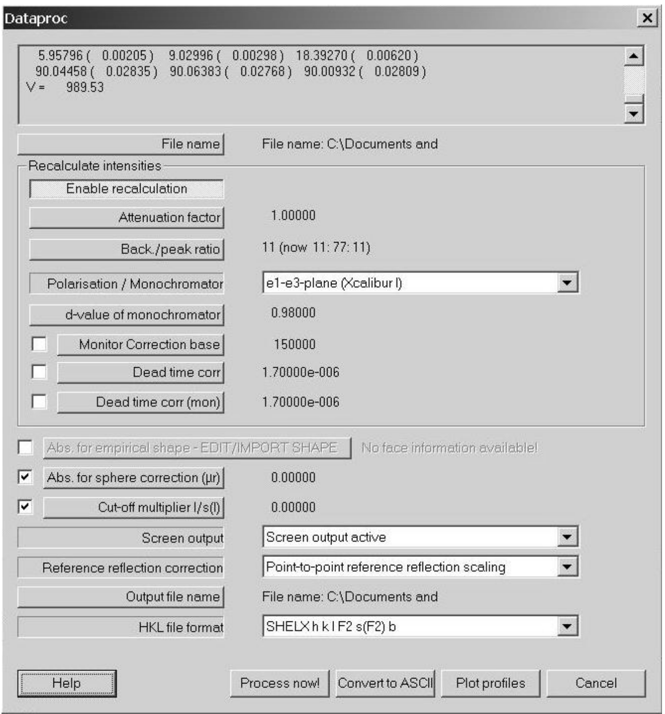



***Figure 4.5 Dataproc programme opened by using the dc redpd command***

# 4.7 Dc Movie - Replay of Data Collection Movie

The CrysAlis software package enables the user to examine the whole of the data collection as a movie. The user can move back and forth through the reflections.

1. Type either **dc redpd** or **dc moviepd** and select the relevant *.dc1 file
2. A control window will appear.
3. Click on the **Play** button and a continuous movie will be played of the data collection. Select forwards or backwards play of the movie by the relevant arrow button. Select the hkl button to see a profile of the reflection with the chosen hkl value.
4. For each reflection, a number of parameters are displayed in the dialogue box: the hkl value, the goniometer angular settings for omega, theta, kappa and phi and the strength of the reflection (ST = strong, WK = weak and LT means not measured).
5. Click on **Exit** to finish data collection movie playback.

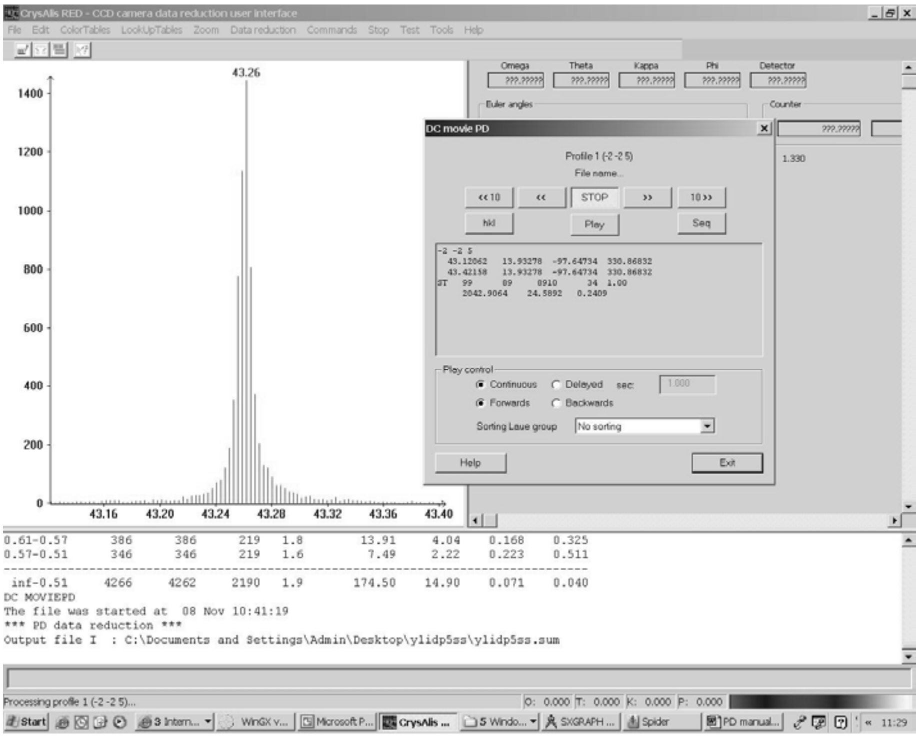



*Figure 4.6 DC MOVIEPD programme*

# 4.8 Absorption Correction

As X-rays pass through the crystal sample, a percentage of the X-rays will be absorbed by the sample. The degree of absorption is related to the distance travelled through the sample and also the composition of the sample. To minimise absorption a spherical crystal is ideal, however, this is often unobtainable. As a result the diffraction data is often corrected for absorption.

The absorption correction incorporated into the CrysAlis software package can be summarised into the following five steps:

1. Record a jpeg movie of the crystal sample
2. Build / modify a 3-D model of the crystal sample
3. Refine / optimise the 3-D model against the X-ray diffraction data
4. Examine 3-D model
5. Repeat steps 2-4 until a satisfactory model and absorption correction have been obtained.

For details please refer to the main Xcalibur operation manual

# 4.9 GRAL - Space Group Determination

The CrysAlis RED, GRAL plug-in wizard guides the user through space group determination. For full details, please refer to the main Xcalibur operator manual.

# 5 Glossary of point detector commands

| Command | Meaning | Example |
|---|---|---|
| abs display | View movie recording and build absorption correction routine | abs display |
| abs grab | Initiate movie recording routine for absorption correction | abs grab |
| af i | Applies an attenuation filter | af i |
| af o | Removes the attenuation filter | af o |
| ce | centre a peak | CE [n\|HP]<br><br>n - setting number:<br><br>n = 0 current angle setting - i.e. do not recover standard setting during refinement<br>n = 1 to 8 standard settings; default – 1<br><br>n = HP (string) carry out centring procedure in 8 symmetrical positions to calculate the crystal displacements from the sphere of confusion of the goniometer as described by A. Ross in Rev. in Miner. & Geochem. **41,** 2000, 559-596 |
| da | detector aperture setting | DA dh dv<br><br>dh = horizontal slit in deg<br>dv = vertical slit in deg |
| dc help | help overview of dc commands | dc help |
| dc applycorrections | Application of data reductions corrections on current frame | dc applycorrections typeofcorrection ([L][P][A][W][S])<br><br>L = lorentz correction.<br>P = polarization correction.<br>A = air path correction.<br>W = window absorption correction.<br>S = scintillator correction. |
| dc hkl | Import hkl file and apply outlier rejection. | dc hkl |
| dc movie | Data collection inspection | dc movie |
| dc visualizecorrections | Visualize data reduction corrections | dc visualizecorrections typeofcorrection ([L][P][A][W][S])scalefactor<br><br>typeofcorrection<br>L = lorentz correction.<br>P = polarization correction.<br>A = air path correction.<br>W = window absorption correction.<br>S = scintillator correction.<br>scalefactor = scale factor with respect to the ideal image 1.0. |
| dc s | Data collection start | dc s |
| dc r | Data collection restart | dc r [[filepath]filename] |
| dc red | Data collection reduction (initiate guided routine) | dc red |
| dc rrp | Data reduction finalization | dc rrp |
| dc unwarp | Reciprocal space reconstruction | dc unwarp |
| dl | speed parameter setting | DL v N<br><br>v [Deg]/[Sec] = phi scanning speed: 0.01 <= v <= 3.0<br>N = discrimination level: 0 < N <= 10000 |
| en | End program and park goniometer | En |
| ga | Gain setting – amplification in the counting chain | GA g<br><br>g = gain for the counting chain. 0.0 - 150.0 |
| gt a | goto angles<br>(omega, theta, kappa, phi) | gt a om th ka ph<br><br>om = omega angle<br>th = theta angle<br>ka = kappa angle<br>ph = phi angle |
| gt d | Moves the CCD camera to a requested distance. | gt d dd<br><br>dd – detector distance in mm |
| gt e | The gt e command has the same meaning as the gt a command, but its parameters are the Euler geometry setting angles (omega, 2theta, chi and phi) instead of kappa geometry setting angles (omega, 2theta, kappa and phi) | GT E ome the chi phi<br><br>ome = omega angle in deg.<br>the = detector angle in deg. |

| | | |
|---|---|---|
| | | chi = chi angle in deg.<br>phi = phi angle in deg. |
| gt o | goto omega | gt o om<br><br>om = omega angle |
| gt t | goto theta angle | gt t th<br><br>th = theta angle |
| gt k | goto kappa angle | gt k ka<br><br>ka = kappa angle |
| gt p | goto phi angle | gt p ph<br><br>ph = phi angle |
| gt r | goto reflection | gt r h k l<br><br>h = h indice<br>k = k indice<br>l = l indice |
| gt chi | goto equivalent omega, theta, kappa, phi values for chi | gt c chi<br><br>chi = chi angle in degrees. |
| gt s | Positions the goniometer at a specified symmetric setting | GT S curset goset<br><br>curset = setting at current pos.<br>goset =  required setting for next move |
| gt help | Help overview of gt commands | gt help |
| gt orient | Initialise guided axial photo routine (once unit cell is known) | gt orient |
| F1 | Activation of the CrysAlis online help | F1 |
| F12 | Activation of the optical alignment menu | F12 |
| gon check | Stadi4 goniometer zero check | gon check |
| gon help | help overview of gon commands | gon help |
| gon init | Xcalibur goniometer initialisation | gon init |
| gon sync | Stadi4 goniometer synchronization | gon sync |
| gon reinit | Xcalibur goniometer re-initialisation | gon reinit |
| hv | high voltage of photomultiplier | HV hv<br><br>hv  =  high voltage for the counting [beam monitor] chain.  KM4: 100.0 - 1000.0 V |
| il | Index limits | Il hmin hmax kmin kmax lmin lmax |
| ll | Low level of the photomultiplier | LL ll [llm]<br><br>ll [llm] = low level for the counting chain. 0.0 - 2.55 |
| ma b | matrix boundary (PD) | MA B (n(1,1)) (n(1,2)) (n(1,3)) (n(2,1)) (n(2,2)) (n(2,3)) (n(3,1)) (n(3,2)) (n(3,3))<br><br>or<br><br>MA B <predef. set no><br><br>n - matrix elements<br>-32 <= n (i,j) <= 32<br><br>predef. set no: The following codes can be selected:<br>5 : 4/mmm<br>7 : -3m1<br>8 : -31m<br>10 : 6/mmm<br>11 : m-3<br>12 : m-3m |
| mo b | mode of background (PD) | MO B (f)<br><br>f - background parameter: 0.01 <= f <= 2.0 . |
| mo s | mode of scan (PD) | MO S m s n1 [n2 Vmin]<br><br>m =  mode of scan:<br><br>0<=m<=2; 0=BPB scan; 1=continuous-integrative step scan; 2=stationary step scan;<br><br>s = number of scan steps (not for m=0): 10<=s<=1024<br>n1 - minimum accepted I/sigma(I) and  n2 = requested I/sigma(I):  -3 <= n1 <= n2 <= 50<br>Vmin - Minimal rescan speed: 0.004768 [Deg]/[Sec] <= Vmin <= 1.5 [Deg]/[Sec] |
| ph e | edit peak hunting table | ph e |

| | | |
|---|---|---|
| ph extractprofiles | extract profiles from data collection | ph extractprofiles |
| ph help | help overview of ph commands | ph help |
| ph s | Peak hunting start | ph s |
| ph reconstruct | reconstruct the peak table with current instrument model | ph reconstruct |
| pt clear | clearing of the peak table | pt clear |
| pt a | add a peak to the peak table | |
| pt e | Peak table edit | pt e |
| pt expand | Peak table expand | pt expand n mmin mmax intmin dmax dmin<br><br>n = number of reflections required<br>mmin = min order of difference -1 for CCD (-3 Point detector)<br>mmax = max order of difference +1 for CCD (+3 Point detector)<br>intmin = Intensity threshold<br>dmax = max d spacing<br>dmin = min d spacing |
| pt l | add reflections to the peak table according to their Laue symmetry | PT L lauecode<br><br>lauecode = The following codes can be selected:<br>1 : -1<br>2 : 2/m<br>3 : mmm<br>4 : 4/m<br>5 : 4/mmm<br>6 : -3<br>7 : -3m1<br>8 : -31m<br>9 : 6/m<br>10 : 6/mmm<br>11 : m-3<br>12 : m-3m |
| pt sa | add reflections from a different setting position | PT SA settingno<br><br>settingno = The settingno refers to the basic measurement settings. |
| rd help | help overview of rd commands | rd help |
| rd jpg | JPEG image | rd jpg [filepath[filename]]<br><br>[[filepath]filename] – Optionally you can put the path and filename on the command line. Note that you have to use quote for filenames with spaces. |
| rd p | Read parameter file | rd p [filepath[filename]]<br><br>[[filepath]filename] – Optionally you can put the path and filename on the command line. Note that you have to use quote for filenames with spaces. |
| rd ph | Read peak hunting table from file | rd ph [filepath[filename]]<br><br>[[filepath]filename] – Optionally you can put the path and filename on the command line. Note that you have to use quote for filenames with spaces. |
| rd t | Read peak table from file | rd t [filepath[filename]]<br><br>[[filepath]filename] – Optionally you can put the path and filename on the command line. Note that you have to use quote for filenames with spaces. |
| rd jpgheader | JPEG image header | rd jpgheader [[filepath]filename.jpg]<br><br>[[filepath]filename] – Optionally you can put the path and filename on the command line. Note that you have to use quote for filenames with spaces. |
| refine model | Refine diffractometer geometry model | refine model |
| refine export | Export details of model refinement to file | refine export |
| rr | reference reflections (PD) | RR rr_num [interval om_tol int_tol [h k l at [h k l at [h k l at]]]]<br><br>rr_num = number of reference reflections: 0 <= rr_num <= 3<br>**if rr_num >0 :**<br>interval = interval between reference reflections measured: 1 <= interval <= 32000<br>om_tol = requested omega repeatability: 0.0025 [Deg] <= om_tol <= 10 [Deg]<br>int_tol = allowed intensity fluctuations parameter (number of sigmas by which a fluctuation triggers the recentering): 1 <= int_tol <= 100<br>h k l = Miller indices of ref. reflection: -126 <= h,k,l <= 126 |

| | | |
|---|---|---|
| | | at = attenuation filter use: 0,1 ( 0 = filter out , 1 = filter in ) |
| scale3 abs | Initialise absorption correction shape optimisation routine | scale3 scale3abs |
| scale3 pack | Initialise scale3pack data scaling plug-in | scale3 scale3pack |
| script | start a script. A script is an ASCII file with the extension *.mac and contains a sequence of CrysAlis commands, which will be, executed one after another. | script [[filepath]filename]<br><br>[[filepath]filename] – Optionally you can put the path and filename on the command line. Note that you have to use quote for filenames with spaces. |
| script help | help overview of script commands | script help |
| sc s | scan speed (PD) | SC S (Vo)<br><br>Vo = scan speed: 0.004768 [Deg]/[Sec] <= Vo <= 1.5 [Deg]/[Sec] |
| sc t | scan type (PD) | SC T (Tx) (f)<br><br>Tx = threshold angle [deg]: 0.0 [Deg] <= Tx <= 90.0 [Deg]<br>f = multiplier for theta motor: 0.0 <= f <= 2.0 |
| sc w | scan width | SC W Oa Ob C<br><br>Oa = scan width (omega): 0.0 [Deg] <= Oa <= 180.0 [Deg]<br>Ob = scan widening parameter: 0.0 [Deg] <= Ob <= 2.0 [Deg]<br>C = scan centre shift parameter: 1.0 <= C <= 1.1 |
| setup detectortype | detector type | setup detectortype |
| setup help | help overview of setup commands | setup help |
| setup options | program options | setup options |
| setup setupfile | start-up file; setup file | setup setupfile |
| sh o | X-ray shutter open | sh o |
| sh c | X-ray shutter closed | sh c |
| sm ao | Single measurement omega scan with angles | sm ao om th ka ph scanwidth time<br><br>om = omega degrees<br>th = theta degrees<br>ka = kappa degrees<br>ph = phi degrees<br>scanwidth = degrees<br>time = seconds |
| sm eta | step scan moving every reflection vertically in the aperture window | SM ETA starteta step steps time<br><br>starteta = starting eta value. Actual goniometer position is assumed to be theta 0.0.<br>step = scanstep in deg.<br>steps = number of steps for scan<br>time = exposure time in sec per step |
| sm help | help overview of sm commands | sm help |
| sm i time | single image photo (static) of exposure time (secs) | sm i time<br><br>time = time in secs |
| sm o | omega scan | sm o startangle scanwidth time |
| sm p | phi scan | sm p startangle scanwidth time |
| sm q | records a q scan | SM Q h k l st #s1 sw1 u1 v1 w1 [#s2 sw2 u2 v2 w2] [filename]<br><br>H K L = indices of selected peak, for which the Omega and Theta angles will be assumed by the program as centre of the scanning area<br>st = measurement time of one scanning point [sec]<br>#s1 = number of scanning points in the [U1 V1 W1] direction<br>sw1 = increment of scanning angle for [U1 V1 W1] direction [Deg]<br>U1 V1 W1 = indices of the first scanning direction<br>#s2 = number of scanning points in the [U2 V2 W2] direction<br>sw2 = increment of scanning angle for [U2 V2 W2] direction [Deg]<br>U2 V2 W2 = indices of the second scanning direction<br>[filename] = optional filename. |
| sm r | measure a single reflection | SM R h k l [psistart [psiend psistep]]<br><br>h k l = reflection index, may be fractional.<br>psistart = optional psi angle.<br>psiend = optional psi start angle for measuring a sequence.<br>psistep = optional psi step angle for measuring a sequence. |
| sm s | single step scan | SM S steps time |

| | | |
|---|---|---|
| | | steps = number of steps for scan<br>time = exposure time in sec per step |
| sm t | theta scan | sm t startangle scanwidth time |
| system dos | Open MSDOS prompt in current directory | system dos |
| system explorer | Open current windows directory | system explorer |
| system help | Help overview of system commands | system help |
| Qvector | Refine incommensurate q vector | qvector mmax crithkl q1 q2 q3 [q1 q2 q3 [q1 q2 q3]] |
| tr | theta range | TR tthmin, tthmax<br><br>tthmin =minimal two-theta<br>tthmax = maximum two-theta<br>0.0 [deg] <= tthmin < tthmax <= 180 [deg] |
| ty help | help overview of ty commands | ty help |
| ty l | Print lattice information to history | ty l |
| ty p | Print parameter file to history | ty p |
| ty t | Print peak table to history | ty t |
| ty imageinfo | Print image information to history | ty imageinfo |
| um crec | change orientation matrix in reciprocal space | um crec [c11 c12 c13 .. c31 c32 c33]<br><br>[c11 c12 c13 .. c31 c32 c33] = reciprocal space transformation matrix |
| um clearskipd | clear skip list | um clearskipd |
| um help | help overview of um commands | um help |
| um hppolynomial | | UM HPPOLYNOMIAL [d0 d1 d2 d3 d4 d5 d6 d7 d8]<br><br>d0-d9 = polynomial coefficients |
| um ip | indexing with peak table printing | um ip [indexrejectioncriterion]<br><br>[indexrejectioncriterion] = rejection criterion which determines the maximum allowed deviation for which a reflection is considered indexed. |
| um ir | indexing with real indices | um ir |
| um overlayskipd | overlay skip list | um overlayskipd |
| um pointdetector | generate goniometer angles from peak table | um pointdetector |
| um r | refine UB under symmetry constraint | um r [symmetrycode]<br><br>[symmetrycode] = The necessary code can be obtained by just typing um r |
| um s | set UB matrix or enter known orientation matrix | um s ub11 ub12 ub13 .. ub31 ub32 ub33 [sub11 sub12 sub13 .. sub31 sub32 sub33]<br><br>ub11 ub12 ub13 .. ub31 ub32 ub33 = orientation matrix<br>[sub11 sub12 sub13 .. sub31 sub32 sub33] = sigma of orientation matrix |
| um shape | View absorption correction model (wire frame less movie overlay) | um shape |
| um showskipd | show skip list | um showskipd |
| um skipd | add item to skip list | um skipd |
| um setqvector | set incommensurate q-vector | um setqvector q1 q2 q3 mmax<br><br>q1 = component of q-vector along a*.<br>q2 = component of q-vector along b*.<br>q3 = component of q-vector along c*.<br>mmax = maximum satellite order. |
| um f | Automatic unit cell determination (indexation, refinement, reduction) | um f [lengthdeviation angledeviation fractionindexed]<br><br>[lengthdeviation angledeviation fractionindexed] = The defaults are 0.05, 0.1, 0.7. You can loosen this condition in case of an unsuccessful indexing: typically 0.3 0.3 0.5. |
| um i | Index and refine unit cell | um i [indexrejectioncriterion]<br><br>[indexrejectioncriterion] = rejection criterion which determines the maximum allowed deviation for which a reflection is considered indexed. |
| um c | Change orientation matrix | um c [change #]\|[c11 c12 c13 .. c31 c32 c33]<br><br>[change #] = transformation number from table obtained by typing um c: [c11 c12 c13 .. c31 c32 c33] – direct space transformation matrix |
| um reduce | Apply Niggli reduction of unit cell | um reduce |
| um r | refine UB under symmetry constraint | um r [symmetrycode]<br><br>[symmetrycode] – The necessary code can be obtained by just typing um r |
| um sarray | Store the defined ub unit cell matrix to a buffer array. 8 available. Used to define a number of | Um sarray arraynum ub11 ub12 ub13 .. ub31 ub32 ub33 |

| | | |
|---|---|---|
| | ub matrices for data reduction of twinned data. | Arraynum (0..7) storage buffers for unit-cell matrices<br>Ub11 … ub33 unit-cell ub matrix |
| um u | activates the procedure to refine the crystal orientation automatically | um u |
| wd i | Write disc image | wd i [[filepath]filename]<br><br>[[filepath]filename] – Optionally you can put the path and filename on the command line. Note that you have to use quote for filenames with spaces. |
| wd flood | Save current image as flood image | wd flood |
| wd t | Write peak table to file | wd t [[filepath]filename]<br><br>[[filepath]filename] – Optionally you can put the path and filename on the command line. Note that you have to use quote for filenames with spaces. |
| wd p | Write parameter file | wd p [[filepath]filename]<br><br>[[filepath]filename] – Optionally you can put the path and filename on the command line. Note that you have to use quote for filenames with spaces. |
| wd cal | Save current parameters into current setup file | wd cal |
| wd help | help overview of wd commands | wd help |
| wd inc | Save current image non-compressed | wd inc [[filepath]filename]<br><br>[[filepath]filename] – Optionally you can put the path and filename on the command line. Note that you have to use quote for filenames with spaces. |
| wd ph | Save peak hunting table (raw profiles) | wd ph [[filepath]filename]<br><br>[[filepath]filename] – Optionally you can put the path and filename on the command line. Note that you have to use quote for filenames with spaces. |
| wd t | Save peak table (indexed xyzs without profiles) | wd t [[filepath]filename]<br><br>[[filepath]filename] – Optionally you can put the path and filename on the command line. Note that you have to use quote for filenames with spaces. |
| wi | window height of the analyser | WI wi [wim]<br><br>wi [wim] = window level for the counting chain: 0.0 - 2.55 |
| zc a | finds the theta zero and the equator/horizon of the machine | ZC A [h k l]<br><br>[h k l]: (optional) Indices of the reflection to use, otherwise the programme will use the current angular setting. |

# Appendix I - High Pressure Crystallography using a DAC on an Xcalibur system

This is intended as a short guide to setting up a DAC for a data collection on an Xcalibur diffractometer. The user is assumed to be familiar with the CrysAlis software and commands and their use for data collections from crystals in air. In the following documentation, commands to be typed into the command line of the Crysalis GUI are indicated thus: **gt r 4 0 0**. Command line entries where numerical values should be substituted are indicated by italics, thus: **gt r *h k l.*** The procedure for different system configurations are outlined, Xcalibur1 and Xcalibur2. Both have interchangeable point detector and area detector.

The Xcalibur 1 diffractometer is equipped with a dual detector arm (see below).

This diffractometer has a dovetail slide for mounting a CCD camera. When the point detector is used for DAC data collections this slide is used to hold a set of additional collimation slits as shown in the picture. The Xcalibur2 system, (and all the other, more recently developed Xcalibur configurations) has a universal theta arm mount which allows easy interchange between point detector and CCD detector.

In addition to the normal equipment provided as part of the Xcalibur diffractometer system you will also need:

- a small spirit level slightly larger than the size of your DAC to align the DAC.
- For the Xcalibur1 system, a dial gauge mounted on a stand so that its axis is horizontal and at the same height as the centre of the goniometer. Used for adjusting the position of the DAC along the beam.

The centering of the DAC may also require the use of the WinIntegrStp software available from /www.crystal.vt.edu/crystal/software/

## I.1 Preparation

Create a filespace for the data collection. Open a file browser and create a new directory for this data (e.g. \P1). Start the CrysAlis program by double-clicking on the desktop icon for CrysAlis CCD

Check that the software is set for point detector operation (GUI should have angles and scan display). If the software is in CCD mode, change to PD mode using the following procedure:

1. Select Tools|Setup file/Xcalibur1PD.par
2. Exit from Tools|Setup
3. Exit from program **(en)**
4. Restart Crysalis CCD from desktop

Switch to DAC mode **(sw s 2)** and set the DAC opening angle to 40deg (or to the value corresponding to your DAC **(sw a *angle*)**.

On the Xcalibur 1 diffractometer, remove the slit assembly from the sled on the dovetail by unscrewing three screws. On the Xcalibur2 diffractometer, ensure that the correct short collimator and long beam stop are installed together with the correct detector limit flag.

# I.2 Physical alignment of DAC

**Xcalibur 1**

Drive the diffractometer to the alignment position **(gt a 0 21.75 0 0).** Load the DAC onto the diffractometer and tighten the base screw firmly. Align the DAC by eye, perpendicular to the beam: Loosen the locking screw for the height adjustment on the goniometer head and rotate the cell until it looks perpendicular to the beam direction. Accurately align the DAC perpendicular to the beam. Slide the sled on the dovetail to the back of the dovetail. Mount the aluminium alignment tool on the sled. Carefully slide the tool in to touch the DAC. Rotate the DAC until the face of the DAC is exactly parallel to the end of the alignment tool. Gently tighten the height locking screw on the goniometer head. Remove the alignment tool. Slide the sled back on the dovetail and lift off the alignment tool, taking care not to hit the beam stop. Set the focus of video microscope and view an image of the cell. Loosen the locking screw of video camera and move it to focus **(gt e 0 0 90 -90).** If image not in focus, adjust the half way to focus with the goniometer head slide, and half with camera adjustment. Repeat until cell is in focus at both of these two positions .Set the height of the DAC **(gt e 0 0 90 90).** Observe the position of gasket hole centre on video screen. **(gt e 0 0 –90 –90).** Compare position of gasket hole and adjust height. Repeat until image of gasket hole does not move vertically between these two positions. Tighten the height locking screw. Check that DAC is still perpendicular to beam at zero **(gt a 0 21.75 0 0)**

See the instructions above for using alignment tool on sled. Correct alignment if necessary. Set cell translation across the beam (x direction) **(gt e 0 0 90 –90).** Observe position of centre of gasket hole **(gt e 0 0 90 90).** Compare position and adjust with slide on goniometer head. Repeat until image of gasket hole does not move between these two positions. Tighten slide locking screw. The DAC may be additionally centred along the beam by use of the beam rocking method described below.

**Xcalibur 2/Xcalibur 3 etc.**

Follow the procedure as above except use the alignment position using the F12 key and the remote pilot. Align the DAC by eye so that it is perpendicular to the beam. Align the aperture in the XY direction using the video microscope and then align along the beam axis as above or use the beam rocking method below.

For systems where the video microscope is aligned vertically, use the following positions to centre the DAC instead of the remote pilot:

**Gt e 0 0 90 90**

**Gt e 0 0 90 -90**

**Gt e 0 0 -90 -90**

**Beam rocking method**

Accurate alignment of a Diamond anvil cell is essential in order to acquire good quality high pressure data. The Xcalibur system allows very easy alignment of the diamond anvil cell along the beam axis using the 'beam rocking' method devised by Ross Angel and Mathias Meyer.

- Flip beamstop out of the way
- Mount the DAC and align the X-Y direction by centring the aperture using the video microscope.
- Record and save images at phi = -30 degrees and +30 degrees (**gt p -30, sm I 0.1, wd I, gt p 30, sm I 0.1, wd i**)
- Subtract one image from the other. (**rd I, ip copy i2 i1, rd I, ip subtract i3 i2 i1**) to observe a difference map of the two images.
- Make small adjustments to the beam axis and repeat. When the alignment is complete, the difference map will be flat.

# I.3 Determine the initial orientation matrix

There are two possibilities, depending on the stage of the high-pressure experiment:

1. The UB from a measurement on the Xcalibur diffractometer at a previous pressure is known
2. The UB from another diffractometer is known
3. The UB is not known.

### I.3.1 UB matrix known from previous measurement

Copy the peak table from the previous measurement. Use the Windows file browser to copy the *.tab file to the current working directory **(rd t).** Or input values into the command line **(um s *u11 u12 u13 u21 u22 u23 u31 u32 u33).*** Check the table is correct by calculating lattice parameters from UB **(ty l).**

### I.3.2 UB matrix unknown

Switch to the CCD camera and perform a short data collection. Find peaks, index and save a peak table. Switch back to the point detector

Go to step I.3.3

### I.3.3 Look for reflections

Insert the pair of slits labeled 3.3 (horizontal) and 2.0 (vertical) with notches towards the back of the cabinet.

| | |
|---|---|
| • Set slit values in software | **da 3.3 2.0** |
| • Drive to a strong reflection | **gt r *h k l*** |
| • Scan the position....set the scan width | **sc w 2.0 0.0** |
| • Do the omega scan | **sm s 40 0.1** |

If the maximum is in the scan, check two more reflections. If all ok, go to step 4. If maximum is not in the scan, drive around in omega and rescan until you find it **(gt o *omega*)** (*omega* should be shifted by 1 deg) **(sm s 40) 0.1.** When a peak is found, drive to position of maximum **(gt o *omega*),** *c*entre the reflection **(ce 0)** and add reflection to peak table **(pt a)**

Look this reflection up in the peak list (pt e). Select it, edit it, insert the correct hkl values, exit the list and save the table **(wd t).** Repeat for another reflection and do two-reflection calculation of UB **(um f2 *a b c* α β γ)**, where ***abc*** and **α β γ** are the estimated cell parameters. Check the result: If the lattice parameters change a lot, then your indexing was incorrect; change the indexing and try again. When you have a valid UB, save it by typing **(wd t).** Check the UB finds other reflections: Drive to a strong reflection (gt r hkl) then see if it is in the detector window **(sm i 1** (or *F7*) and observe counts)

## I.4 Refine UB

Enter the peak table window by typing pt e. Make a note of the *hkl* and then delete all reflections (see note at end of this table). Insert the *hkl* of one of each symmetry-equivalent set

Exit from the peak table editor and expand the peak list by Laue symmetry **(pt l *n*)** (if you do not know ***n*** for your Laue group then type **pt l** to obtain a list). At this stage you need 20-30 strong reflections **(pt e).** Edit the peak list to remove reflections with low kappa angle:

- Select “angles” at bottom of display
- Click on “kappa” column header to order reflections
- Delete reflections with $-15^{o} < \kappa < 15^{o}$.
- Exit the peak list editor
- Save the peak list **(wd t)**
- Start reflection centering **(um u)**

*Note:* If a reflection in the list cannot be centered it will be skipped and deleted from the list. If many reflections are skipped then either the UB matrix and the cell parameters are wrong, or the software has the wrong values loaded (with **da**) for the detector slits.

To recover from this problem, read the original table back into software, **rd t.** Enter the correct slit values **da *hslit vslit*** and repeat centering **um u.** At the end of centering, the UB is determined. Save it and the peak positions **wd t.** Switch to smaller slits, h=2.0, v=1.0 and update values in software: **da 2.0 1.0.** Check the UB finds reflections with smaller slits: Drive to a strong reflection and see if it is in the detector window **gt r *h k l.*** Record a still image **sm i 1** (or *F7*) and observe counts. If ok, repeat centering **um u.** At the end of centering, the UB is determined. Save it and the peak positions **wd t**

**Note on the peak table: *The Crysalis centering procedure um u works by first driving to the angular positions given in the peak table. This is different from the Single software in which the starting position for centering is calculated from the* hkl *in the peak table, and the current UB matrix. This means that in Crysalis when the UB is changed significantly, the peak table must be cleared and the indices of reflections be reloaded into the table; this procedure ensures that the peak positions are calculated from the current UB.***

# I.5 Determine crystal offsets

At this stage the gasket hole of the DAC has been well-centered optically across the beam, but the positioning along the beam has relied on focusing the video microscope on the sample. The centering along the beam can be improved by “diffracted beam centering”. There are two ways to achieve this:

1. By 8-position centering of a single reflection with a Eulerian-chi value between $80^{o}$ and $90^{o}$.
2. By collecting data scans of 30 or more low-angle reflections and refining the crystal offsets by the method of Dera and Katrusiak (1999, Journal of Applied Crystallography 32:510-515).

Method 1 takes less time, but method 2 is often more reliable. Both alternatives are described below:

**Method 1 - 8-position centering**

- Find a strong reflection with Eulerian chi > 80$^{o}$ **gt r *h k l***
- Take a still image **sm i 1**
- Do 8-position centering **ce HP**
- Adjust goniometer position according to offsets from **ce HP** procedure
- Repeat until offsets are small or zero

**Method 2 - Crystal offsets from data collection**

Set up parameters for a short low-angle data collection, as follows.

| | |
|---|---|
| Set detector slits to h=2.0, v=0.5 and exchange the brass slits on the detector | |
| Update values in software: | **da 2.0 0.5** |
| Set scan parameters to stop rescanning | **mo s 1 60 10 0 0.005** |
| Set fast scan speed | **sc s 0.05** |
| Set background calc | **mo b 0.5** |
| Set scan width | **sc w 1.200 0.000 1.00300** |
| Set omega scan | **sc t 0.0 0.0** |
| Set index limits to cover all reciprocal space *Note:* this sets the maximum values of indices to be tested against 2theta limits etc. Just make them sufficiently large | **il –10 10 –10 10 –10 10** |
| Clear ma limits | **ma b 0 0 0 0 0 0 0 0 0** |
| Set 2theta limits | **tr 0 25** |
| Set absence conditions | **rc** |
| Clear reference reflections | **rr 0** |
| Check all values are correct | **ty p** |
| Check number of reflections that will be collected | **dc t** |
| Adjust 2theta limits until you have 30-50 reflections to be collected | **tr *tthmin tthmax*** |

Save parameters (it is useful to call this something like *orient.par*) **wd p**

Start data collection (use a distinct filename such as orient*n*) where *n* indicates the iteration through this process **dc s**

When data collection is complete, export the data to a *dca* file

Open the Crysalis Reduce software and type **dc redpd** on its command line. Select your data file and convert to Ascii. Open the WinIntegrStp program by double-clicking on desktop icon. Select the *dca* file you just created.

Select Xcalibur.par as the instrument parameter file. Run preprocessing option to obtain the peak positions

- Set I/sigma to 10.0
- Set Intensity, peak width, position to be refined
- Set background to not refined, with default value "D"
- Set eta and Iratio to not refined
- Start preprocess with "Go"

If insufficient (<20) reflections are stored after Preprocess, reduce I/sigma or adjust the test limits on the parameters. Once you have >20 reflections stored from Preprocess, calculate the UB (Utilities|Calc UB). Select refine crystal offsets and run. Record the crystal offsets reported (in mm). The X and Z offsets should already be small (<50micron). The Y offset is along the beam. If it is less than 30 micron go to step I.3.6. If Y offset >30 micron proceed as follows:

- Drive goniometer to zero **gt a 0 0 0 0**
- Place the dial gauge in contact with the downstream face of the cell.
- Adjust the cell position along the beam   One division on the dial gauge is 25 micron.
- If the Y offset is positive, move the DAC towards the X-ray tube.
- If the Y offset is negative, move the DAC away from the X-ray tube.
- Repeat step 5.2 until Y offset is <30 micron.

# I.6 Data Collection

Re-determine the UB matrix:

Exchange brass slits on detector. Update values in software: **da 1.33 0.5** then **um u.** Save peak table to disk **wd t.** On the Xcalibur 1 system, Install the additional slits on detector arm. Screw down the slits onto the carrier on the dovetail. Set the slide to 9.55. Set the detector slits to h=2.0, v=0.5. Exchange the brass slits on detector and update values in software: **da 2.0 0.5** Set up parameters for the data collection as follows:

| | |
|---|---|
| Set scan parameters | **mo s 1 60 1 10 0.005** |
| Set fast scan speed | **sc s 0.05** |
| Set background calc | **mo b 0.5** |
| Set scan width | **sc w 1.200 0.000 1.00300** |
| Set omega scan | **sc t 0.0 0.0** |
| Set index limits to cover required portion of reciprocal space*Note:* this sets the maximum values of indices to be tested against 2theta limits etc. Just make them sufficiently large. | **il *hmin hmax kmin kmax lmin lmax*** |
| Set ma limits if required | **ma b *n*** |
| Set 2theta limits | **tr *thmin thmax*** |
| Set absence conditions | **rc** |
| Set reference reflections | **rr 3 200 0.15 15.0 *h k l 0 h k l 0 h k l 0*** |
| Check all values are correct | **ty p** |
| Check number of reflections to be collected | **dc t** |
| Check that parameters and UB are ok by scanning several reflections | **sm r *h k l*** |
| Save parameters | **wd p** |
| Start data collection | **dc s** |

# I.6 When Data Collection Is Completed

- Open the Crysalis Reduce software
- type **dc redpd** on its command line
- Select your data file
- Select Convert to Ascii
- Open the WinIntegrStp program by double clicking on desktop icon
- Select the *dca* file you just created

- Select Xcalibur.par as the instrument parameter file.
- Check the scans are ok and centered; Use Integrate | Manual Profile Fit to review the dataset
- Integrate the data and refine the structure